Supporting Information

Total Synthesis of Zincophorin Methyl Ester

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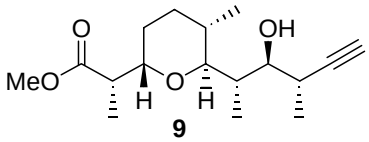
General

TLC was performed on Merck $60F_{254}$ silica gel plates and visualized either with a UV lamp (254 nm), or by using a solution of *p*-anisaldehyde/sulfuric acid/acetic acid in EtOH followed by heating.

Flash chromatography was performed with SDS 60 silica gel (230-400 mesh).

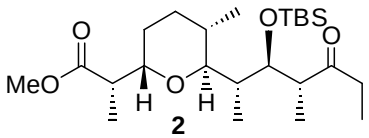
Infrared (IR) spectra were recorded on a Perkin-Elmer 298, wavenumbers are indicated in cm^{-1}. ^{1}H NMR spectra were recorded on a Bruker AC 300 at 300 MHz and data are reported as follows: chemical shift in ppm from tetramethylsilane as an internal standard, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of non equivalent resonances), integration. ^{13}C NMR spectra were recorded on a Bruker AC 300 at 75 MHz and data are reported as follows: chemical shift in ppm from tetramethylsilane with the solvent as an internal indicator ($CDCl_3$ δ 77.0 ppm), multiplicity with respect to proton (deduced from DEPT experiments, s = quaternary C, d = CH, t = CH_2, q = CH_3). Mass spectra with electronic impact (MS-EI) were recorded from a Hewlett-Packard tandem 5890A GC (12 m capillary column) – 5971 MS (70 eV). Mass spectra with chemical ionization (MS-CI^+) and high resolution mass spectra (HRMS) were performed by the Centre de Spectrochimie Organique de l'Ecole Normale Supérieure Ulm (Paris). Elemental analyses were performed by the Centre Régional de Microanalyses (Université Pierre et Marie Curie, Paris VI).

Spectral data of key intermediates



9

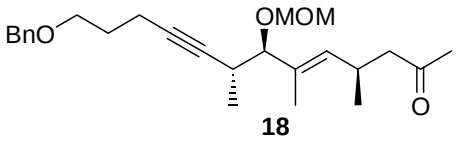
Methyl (2*S*)-2-[(2*S*,5*S*,6*S*)-6-((1*S*,2*S*,3*S*)-2-hydroxy-1,3-dimethylpent-4-ynyl)-5-methyltetrahydro-2*H*-pyran-2-yl]propanoate (9): White solid, R_f 0.31 (petroleum ether/EtOAc: 80/20); m.p. = 90 °C; $[\alpha]_D^{20}$ +44.2 (*c* 1.15, $CHCl_3$); IR (film): 3530, 1710, 1275, 1255, 1245, 1235, 1105, 1080, 1040, 1020, 970 cm^{-1}; ^1H NMR ($CDCl_3$) δ 3.94 (m, 1H), 3.72 (s, 3H), 3.67 (dd, *J* = 9.9 and 1.8 Hz, 1H), 3.49 (d, *J* = 6.6 Hz, 1H, OH), 3.22 (m, 1H), 3.17 (dq, *J* = 11.0 and 6.8 Hz, 1H), 2.67 (m, 1H), 2.05 (d, *J* = 2.2 Hz, 1H), 1.93 (m, 1H), 1.82-1.54 (m, 3H), 1.29 (d, *J* = 7.0 Hz, 3H), 1.30-1.21 (m, 2H), 1.06 (d, *J* = 6.6 Hz, 3H), 0.83 (d, *J* = 6.3 Hz, 3H), 0.82 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 176.7 (s), 85.4 (s), 75.3 (d), 74.7 (d), 73.8 (d), 69.6 (d), 52.0 (q), 39.8 (d), 38.2 (d), 31.8 (d), 29.2 (d), 27.5 (t), 25.3 (t), 18.5 (q), 17.6 (q), 14.2 (q), 9.2 (q); MS-CI^+ (CH_4) *m/z* (%): 297 ($M+H^+$, 100), 279 (13), 243 (23), 185 (35); HRMS (CI^+, CH_4) Calcd for $C_{17}H_{29}O_4$ ($M+H^+$): 297.2068. Found: 297.2065.



2

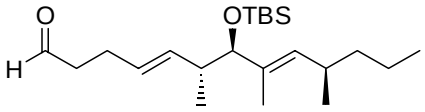
Methyl (2*S*)-2-((2*S*,5*S*,6*S*)-6-{[(1*R*,2*R*,3*R*)-2-(*tert*-butyldimethylsilyl)oxy]-1,3-dimethyl-4-oxohexyl}-5-methyltetrahydro-2*H*-pyran-2-yl)propanoate (2): Colorless oil, $[\alpha]_D^{20}$ -10.8 (*c* 0.65, $CHCl_3$); IR (film) 1740, 1720, 1255, 1165, 1050, 835, 775 cm^{-1}; ^1H NMR ($CDCl_3$) δ 3.93 (dd, *J* = 7.0 and *J* = 4.0 Hz, 1H), 3.71 (s, 3H), 3.70 (m, 1H), 3.43 (dd, *J* = 8.5 and 3.7 Hz, 1H), 2.77 (apparent quintet, *J* = 7.0 Hz, 1H), 2.59 (dq, *J* = 8.8 and 7.0 Hz, 1H), 2.51 (q, *J* = 7.2 Hz, 2H), 2.13 (m, 1H), 1.87-1.72 (m, 2H), 1.54-1.38 (m, 3H), 1.08 (d, *J* = 7.0 Hz, 3H), 1.03 (d, *J* = 7.0 Hz, 3H), 1.01 (t, *J* = 7.2 Hz, 3H), 0.97 (d, *J* = 7.4 Hz, 3H), 0.89 (d, *J* = 7.0 Hz, 3H), 0.87 (s, 9H), 0.10 (s, 3H), 0.01 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 213.7 (s), 175.8 (s), 78.8 (d), 75.9 (d), 71.8 (d), 51.6 (q), 49.2 (d), 44.8 (d), 39.2 (d), 36.6 (t), 28.1 (d), 25.9 (q, 3C), 25.5 (t), 23.6 (t), 18.2 (q), 18.0 (s), 13.6 (q), 13.4 (q), 9.6 (q), 7.4 (q), -4.3 (q), -4.8 (q); MS-

CI^+ (CH_4) *m/z* (%): 443 ($M+H^+$, 100), 427 (7), 385 (8), 311 (30), 243 (8), 185 (32); HRMS (CI^+, CH_4) Calcd for $C_{24}H_{47}O_5Si$ ($M+H^+$): 443.3193. Found: 443.3185.



18

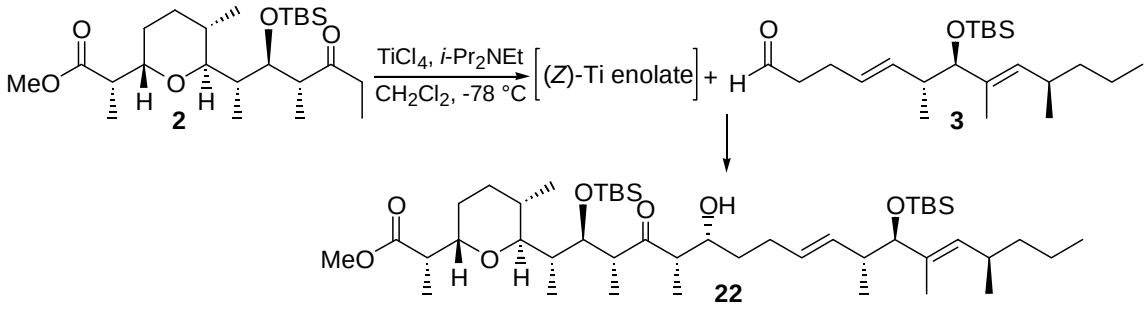
(*E*)-(4*R*,7*R*,8*R*)-13-Benzyloxy-7-methoxymethoxy-4,6,8-trimethyltridec-5-en-9-yn-2-one (18): Colorless oil; R_f 0.50 (petroleum ether/EtOAc: 70/30); $[\alpha]_D^{20}$ +1.1 (*c* 1.15, $CHCl_3$); IR (film) 1715, 1150, 1095, 1030, 920, 740, 700 cm^{-1}; ^1H NMR ($CDCl_3$) δ 7.35-7.25 (m, 5H), 5.18 (br d, *J* = 9.6 Hz, 1H), 4.61 (d, *J* = 6.6 Hz, 1H), 4.51 (d, *J* = 6.6 Hz, 1H), 4.50 (s, 2H), 3.71 (d, *J* = 9.2 Hz, 1H), 3.55 (t, *J* = 6.4 Hz, 2H), 3.41 (s, 3H), 2.99 (m, 1H), 2.61 (m, 1H), 2.38 (d, *J* = 7.0 Hz, 2H), 2.29 (td, *J* = 7.2 and 2.2 Hz, 2H), 2.09 (s, 3H), 1.79 (m, 2H), 1.56 (d, *J* = 1.5 Hz, 3H), 0.99 (d, *J* = 7.0 Hz, 3H), 0.95 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 207.7 (s), 138.6 (s), 136.2 (d), 131.2 (s), 128.3 (d, 2C), 127.6 (d, 2C), 127.5 (d), 93.0 (t), 84.9 (d), 83.1 (s), 80.2 (s), 72.9 (t), 69.1 (t), 55.4 (q), 50.7 (t), 30.5 (q), 29.2 (t), 29.1 (d), 28.6 (d), 20.8 (q), 18.0 (q), 15.7 (t), 10.8 (q); MS-EI *m/z* (%): 339 (M^+-OMOM, 0.4), 200 (13), 199 (100), 141 (16), 137 (29), 125 (34), 121 (12), 109 (25), 95 (12), 91 (67). Anal Calcd for $C_{25}H_{36}O_4$: C, 74.96 ; H, 9.06. Found: C, 74.79 ; H, 9.25.



3

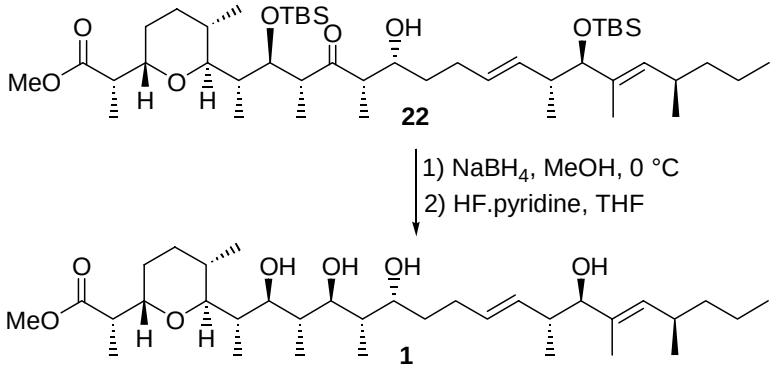
(4*E*,6*R*,7*R*,8*E*,10*R*)-7-[(*tert*-Butyldimethylsilyl)oxy]-6,8,10-trimethyltridecadi-4,8-enal (3): Colorless oil; $[\alpha]_D^{20}$ -11.8 (*c* 0.93, $CHCl_3$); IR (film) 1725, 1250, 1060, 870, 835, 775 cm^{-1}; ^1H NMR ($CDCl_3$) δ 9.78 (t, *J* = 1.8 Hz, 1H), 5.48 (dd, *J* = 15.4 and 7.2 Hz, 1H), 5.37 (m, 1H), 4.99 (br d, *J* = 9.6 Hz, 1H), 3.56 (d, *J* = 8.5 Hz, 1H), 2.50 (m, 2H), 2.44-2.31 (m, 3H), 2.22 (m, 1H), 1.53 (d, *J* = 1.5 Hz, 3H), 1.32-1.11 (m, 4H), 0.91 (d, *J* = 6.6 Hz, 3H), 0.89-0.82 (m, 3H), 0.86 (s, 9H), 0.79 (d, *J* = 6.6 Hz, 3H), -0.01 (s, 3H), -0.05 (s, 3H); ^{13}C RMN ($CDCl_3$) δ 202.3 (s), 135.6 (d), 134.7 (s), 134.3 (d), 126.8 (d), 83.7 (d), 43.5 (t), 40.8 (d), 39.9 (t), 31.7 (d), 25.8 (q, 3C), 25.3 (t), 20.7 (q), 20.6 (t), 18.2 (s), 17.0 (q), 14.2 (q), 11.1 (q), -4.5 (q), -5.0 (q); MS-CI^+ (CH_4) *m/z* (%): 367 ($M+H^+$, 10), 351 (20), 309 (13), 255 (56),

235 (M^+-OTBS, 100), 217 (17), 151 (22), 111 (21); HRMS (CI^+, CH_4) Calcd $C_{22}H_{43}O_2Si$ ($M+H^+$): 367.3032. Found: 367.3029.



Methyl (2*S*)-2-((2*S*,5*S*,6*S*)-6-{(1*R*,2*R*,3*R*,5*S*,6*R*,9*E*,11*R*,12*R*,13*E*,15*R*)-2,12-bis[(*tert*-butyldimethylsilyl)oxy]-6-hydroxy-1,3,5,11,13,15-hexamethyl-4-oxooctadi-9,13-enyl}-5-methyltetrahydro-2*H*-pyran-2-yl)propanoate (22): To a solution of ethylketone **2** (60.0 mg, 0.136 mmol) in anhydrous CH_2Cl_2 (2.5 mL) at -78 °C, was added a solution of freshly distilled $TiCl_4$ (190 μL, 0.75 M in CH_2Cl_2, 0.142 mmol, 1.05 equiv.) and after 1 min, *i*-Pr_2NEt (30 μL, 0.17 mmol, 1.3 equiv.) was added. After 1 h at -78 °C, a solution of aldehyde **3** (36 mg, 0.098 mmol, 0.72 equiv.) in CH_2Cl_2 (2.5 mL) was added dropwise and the reaction was quenched 2 h later by addition of a saturated aqueous solution of NH_4Cl. The reaction mixture was diluted with Et_2O and H_2O, the layers were separated and the aqueous phase was extracted with ether. The combined extracts were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The ^1H NMR spectrum of the crude material indicated the formation of **22** with high diastereoselectivity (d.r. > 96/4). Purification by flash chromatography (petroleum ether/Et_2O: 90/10, 85/15) afforded 55 mg (70%) of **22** as a colorless oil and 18 mg (30%) of ethylketone **2** was recovered; $[\alpha]_D^{20}$ -21.3 (*c* 1.15, $CHCl_3$); IR (film) 3440, 1740, 1695, 1255, 1165, 1060, 835, 775 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.45 (dd, *J* = 15.4 and 6.2 Hz, 1H), 5.36 (m, 1H), 4.97 (br d, *J* = 8.5 Hz, 1H), 4.04 (m, 1H), 3.90 (dd, *J* = 7.0 and 3.3 Hz, 1H), 3.71 (s, 3H), 3.70 (m, 1H), 3.55 (d, *J* = 8.1 Hz, 1H), 3.42 (dd, *J* = 7.9 and 3.9 Hz, 1H), 3.13 (br s, 1H, OH), 3.03 (apparent quintet, *J* = 7.0 Hz, 1H), 2.67-2.57 (m, 2H), 2.36 (m, 1H), 2.23-2.11 (m, 3H), 1.99 (m, 1H), 1.87-1.70 (m, 2H), 1.65-1.39 (m, 4H), 1.53 (d, *J* = 1.5 Hz, 3H), 1.35-1.19 (m, 5H), 1.13 (d, *J* = 7.4 Hz, 3H), 1.08 (d, *J* = 7.0 Hz, 3H), 1.01 (d, *J* = 6.6 Hz, 3H), 0.97 (d, *J* = 7.0 Hz, 3H), 0.93 (d, *J* = 7.0 Hz, 3H), 0.90 (d, *J* = 7.0 Hz, 3H), 0.87 (s, 9H), 0.88-0.84 (m, 3H), 0.85 (s, 9H), 0.78 (d, *J* = 7.0 Hz, 3H), 0.09 (s, 3H), 0.01 (s, 3H), -0.02 (s, 3H), -0.06 (s, 3H); ^{13}C RMN ($CDCl_3$) δ 219.0 (s), 175.7 (s), 134.8 (s),

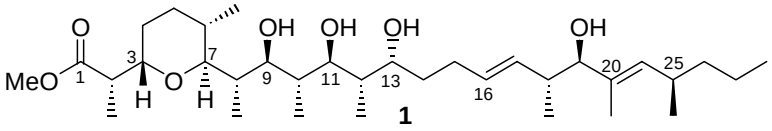
134.5 (d), 134.1 (d) 128.7 (d), 83.9 (d), 78.7 (d), 76.7 (d), 72.0 (d), 69.7 (d), 51.6 (q), 50.4 (d), 46.7 (d), 44.4 (d), 40.8 (d), 40.0 (d), 39.9 (t), 33.6 (t), 31.6 (d), 29.4 (t), 28.3 (d), 25.94 (q, 3C), 25.87 (q, 3C), 25.6 (t) 23.7 (t), 20.8 (q), 20.7 (t), 18.2 (q), 18.1 (s, 2C), 17.0 (q), 14.23 (q), 14.16 (q), 13.5 (q), 11.1 (q), 9.3 (q), 8.7 (q), -4.4 (q), -4.5 (q, 2C), -4.9 (q); HRMS (CI^+, NH_3) Calcd $C_{46}H_{92}NO_7Si_2$ ($M+NH_4^+$): 826.6412. Found: 826.6417.



Methyl (2*S*)-2-[(2*S*,5*S*,6*S*)-6-((1*S*,2*S*,3*S*,4*S*,5*S*,6*R*,9*E*,11*R*,12*R*,13*E*,15*R*)-2,4,6,12-tetrahydroxy-1,3,5,11,13,15-hexamethyloctadi-9,13-enyl)-5-methyltetrahydro-2*H*-pyran-2-yl]propanoate (zincophorin methyl ester) (1): To a solution of **22** (23 mg, 0.028 mmol) in MeOH (5 mL) at 0-5°C, was added portionwise $NaBH_4$ (40 mg, 0.11 mmol, 4 equiv.) [four portions, every 20 min]. After 1 h, the reaction mixture was hydrolyzed with a saturated aqueous solution of Rochelle's salt and diluted with H_2O and Et_2O. The layers were separated and the aqueous phase was extracted with Et_2O. The combined extracts were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was dissolved in THF (5 mL) and to the resulting solution at 0-5 °C [polyethylene container], was added HF.pyridine complex (1 mL). After 1 h 30 at rt, the reaction mixture was diluted with Et_2O and H_2O, cautiously neutralized by addition of solid $NaHCO_3$ and extracted with Et_2O. The combined extracts were dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude material was purified by flash chromatography (*n*-hexane/EtOAc: 50/50) to afford 11 mg (66%) of zincophorin methyl ester **1**; R_f 0.6 (*n*-hexane/EtOAc: 50/50); R_f 0.4 (C_6H_6/Et_2O: 50/50); $[\alpha]_D^{20}$ +21.3 (*c* 0.4, $CHCl_3$); IR ($CHCl_3$) 3380, 1730, 1460, 1380, 1280, 1260, 1215, 1120, 1080, 1020, 970 cm^{-1}; ^1H NMR ($CDCl_3$) δ 5.93 (s, 1H), 5.61 (dt, *J* = 15.1 and 6.6 Hz, 1H), 5.34 (dd, *J* = 15.1 and 8.8 Hz, 1H), 5.11 (br d, *J* = 9.2 Hz, 1H), 4.43 (d, *J* = 8.1 Hz, 1H), 4.12-4.06 (m, 3H), 3.76 (d, *J* = 10.3 Hz, 1H), 3.72 (s, 3H), 3.63 (dd, *J* = 8.8 and

1.8 Hz, 1H), 3.55 (d, *J* = 9.6 Hz, 1H), 3.44 (m, 1H), 3.23 (apparent dq, *J* = 10.8 and 7.0 Hz, 1H), 2.41 (m, 1H), 2.29-2.14 (m, 3H), 2.12 (br s, 1H), 2.08-1.96 (m, 2H), 1.78-1.52 (m, 4H), 1.60 (d, *J* = 1.5 Hz, 3H), 1.41-1.15 (m, 6H), 1.10 (d, *J* = 6.6 Hz, 3H), 1.08 (d, *J* = 6.6 Hz, 3H), 1.06 (d, *J* = 7.0 Hz, 3H), 0.94 (d, *J* = 6.6 Hz, 3H), 0.90-0.80 (m, 5H), 0.84 (d, *J* = 6.6 Hz, 3H), 0.82 (d, *J* = 6.2 Hz, 3H), 0.66 (d, *J* = 6.6 Hz, 3H); ^{13}C RMN ($CDCl_3$) δ 175.6 (s), 135.7 (d), 133.4 (d), 133.3 (s), 133.2 (d), 84.4 (d), 84.0 (d), 81.8 (d), 76.1 (d), 74.5 (d), 68.9 (d), 52.3 (q), 41.8 (d), 39.9 (t), 39.7 (d), 38.4 (d), 37.5 (d), 34.4 (t), 34.0 (d), 31.8 (d), 31.6 (d), 29.1 (t), 26.3 (t), 25.0 (t), 21.0 (q), 20.6 (t), 17.7 (q), 17.5 (q), 14.8 (q), 14.2 (q), 13.3 (q), 11.25 (q), 11.20 (q), 10.8 (q); HRMS (CI^+, NH_3) Calcd for $C_{34}H_{63}O_7$ ($M+H^+$): 583.4574. Found: 583.4578.

Comparison with the literature data reported for **1** (Danishefsky et al. *J. Am. Chem. Soc.* **1988**, *110*, 4368)

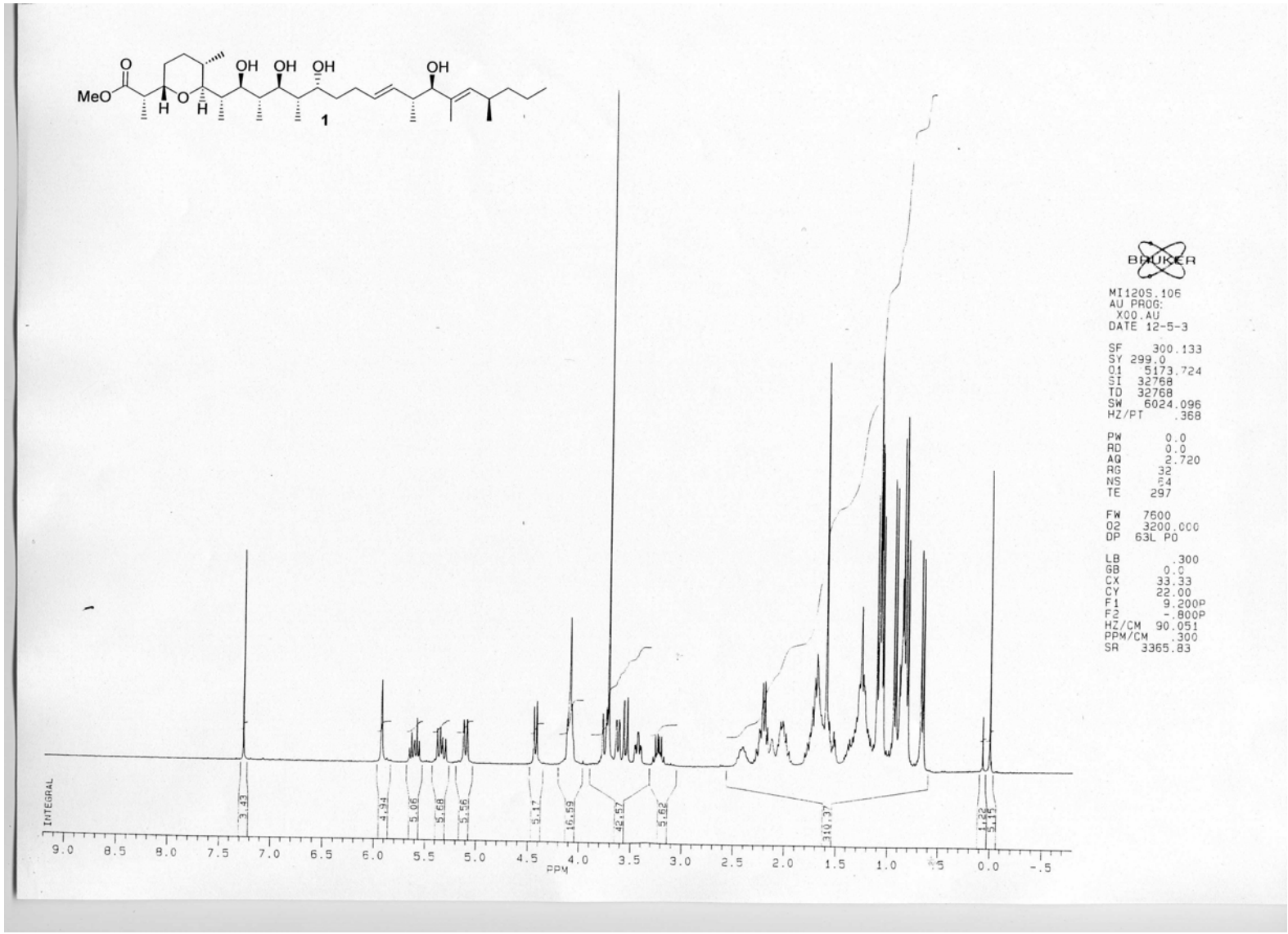


1

Observed data for **1**	Literature data reported for **1**
^{1}H NMR (300 MHz, $CDCl_3$) δ	^{1}H NMR (500 MHz, $CDCl_3$) δ
5.93 (s, 1H)	5.93 (s, 1H)
5.61 (dt, *J* = 15.1 and 6.6 Hz, 1H)	5.62 (apparent dt, *J* = 15.0 and 3.2[a] Hz, 1H)
5.34 (dd, *J* = 15.1 and 8.8 Hz, 1H)	5.35 (dd, *J* = 15.0 and 9.0 Hz, 1H)
5.11 (br d, *J* = 9.2 Hz, 1H)	5.11 (d, *J* = 9.3 Hz, 1H)
4.43 (d, *J* = 8.1 Hz, 1H)	4.43 (d, *J* = 8.1 Hz, 1H)
4.12-4.06 (m, 3H)	4.1 (m, 3H)
3.76 (d, *J* = 10.3 Hz, 1H)	3.76 (d, *J* = 10.1 Hz, 1H)
3.72 (s, 3H)	3.73 (s, 3H)
3.63 (dd, *J* = 8.8 and 1.8 Hz, 1H)	3.63 (dd, *J* = 8.6 and 1.7 Hz, 1H)
3.55 (d, *J* = 9.6 Hz, 1H)	3.56 (d, *J* = 9.2 Hz, 1H)
3.44 (m, 1H)	3.44 (apparent dt, *J* = 9.0 and 1.8 Hz, 1H)
3.23 (apparent dq, *J* = 10.8 and 7.0 Hz, 1H)	3.23 (apparent dq, *J* = 10.8 and 7 Hz, 1H)
2.41 (m, 1H)	2.42 (m, 1H)
2.29-2.14 (m, 3H)	2.22 (m, 3H)
2.12 (br s, 1H)	2.12 (br s, 1H)
2.08-1.96 (m, 2H)	2.01 (m, 2H)
1.78-1.52 (m, 4H)	1.75 (m, 4H)
1.60 (d, *J* = 1.5 Hz, 3H)	1.6 (d, *J* = 1.3 Hz, 3H)
1.41-1.15 (m, 6H)	1.3 (m, 6H)
1.10 (d, *J* = 6.6 Hz, 3H)	1.11 (d, *J* = 7.0 Hz, 3H)
1.08 (d, *J* = 6.6 Hz, 3H)	1.10 (d, *J* = 6.6 Hz, 3H)
1.06 (d, *J* = 7.0 Hz, 3H)	1.07 (d, *J* = 8.0 Hz, 3H)
0.94 (d, *J* = 6.6 Hz, 3H)	0.95 (d, *J* = 6.6 Hz, 3H)
0.90-0.80 (m, 5H)	0.88 (m, 5H)
0.84 (d, *J* = 6.6 Hz, 3H)	0.85 (d, *J* = 6.8 Hz, 3H)
0.82 (d, *J* = 6.2 Hz, 3H)	0.82 (d, *J* = 6.6 Hz, 3H)
0.66 (d, *J* = 6.6 Hz, 3H)	0.67 (d, *J* = 7.0 Hz, 3H)

(a) The surprisingly low value (3.2 Hz) indicated for the corresponding $J^3(H_{16(vinylic)}\text{-}H_{15(allylic)})$ coupling constant is not in agreement with the one measured on our spectrum of zincophorin methyl ester (6.6 Hz). However the reported value appears to be abnormal for such couplings and is likely to be a mistake in the listing of the spectroscopic data.

Copies of the ^{1}H and ^{13}C NMR spectra of zincophorin methyl ester **1**: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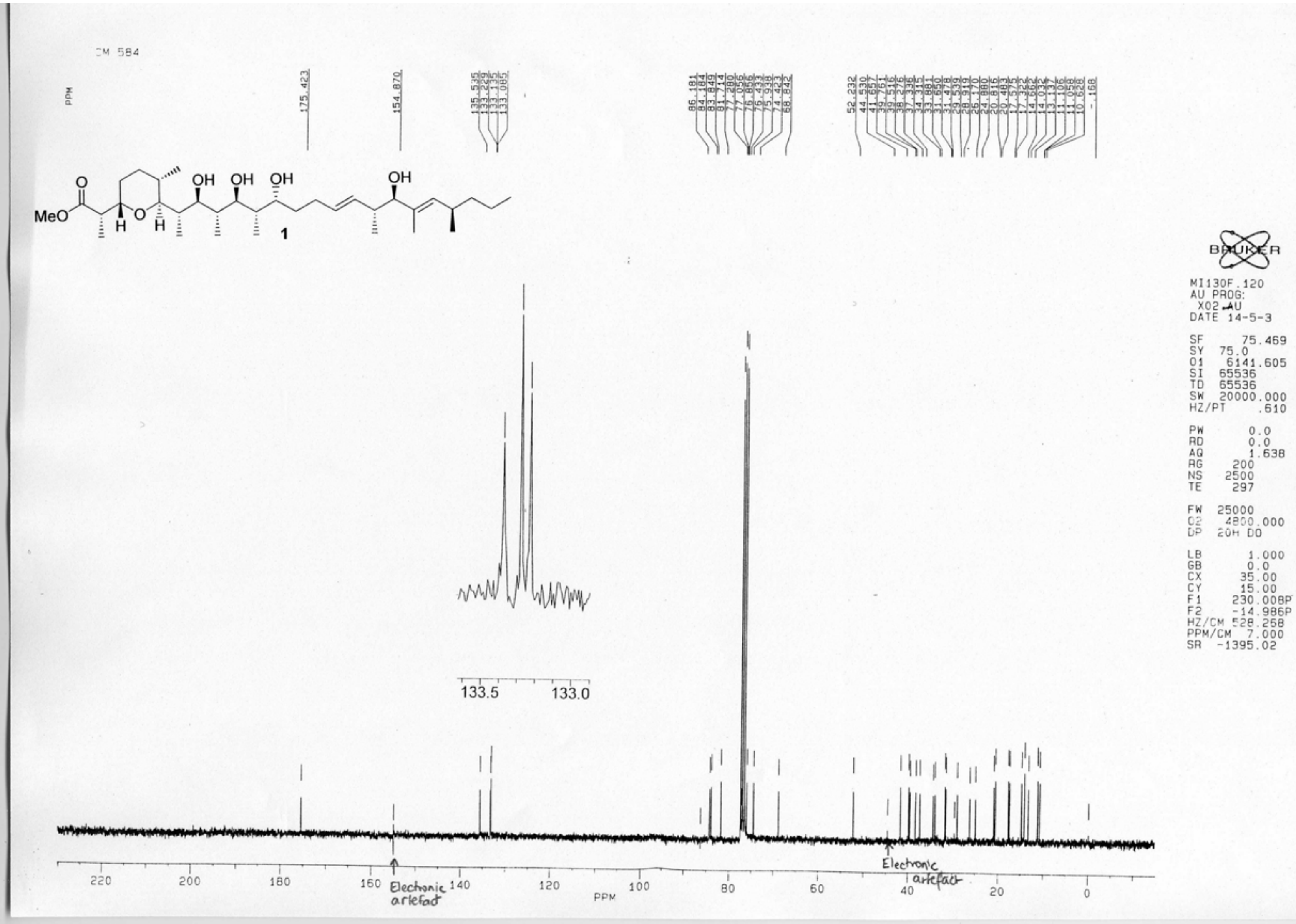
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